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“Step Out From the Old to the New”

IS 3123 (2005): Hydroxycitronellal [PCD 18: Natural and Synthetic Fragrance Materials]



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Bhartrhari—Nitiśatakam

“Knowledge is such a treasure which cannot be stolen”

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भारतीय मानक
हाइड्रॉक्सीसिट्रोनीलल — विशिष्टि
(तीसरा पुनरीक्षण)

Indian Standard
HYDROXYCITRONELLAL — SPECIFICATION
(*Third Revision*)

ICS 71.100.60

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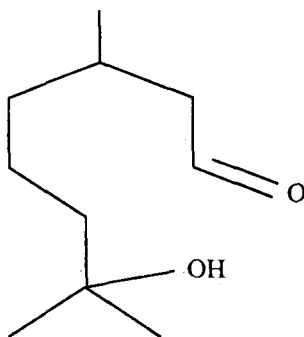
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FOREWORD

This Indian Standard (Third Revision) was adopted by the Bureau of Indian Standards, after the draft finalized by the Natural and Synthetic Fragrance Materials Sectional Committee had been approved by the Petroleum, Coal and Related Products Division Council.

This standard was first published in 1965 and revised in 1980 and 1993 respectively. In the present revision, the purity has been revised to 98 percent to be in line with the international specifications. Requirements for description, odour and acid value have been revised. Requirement for higher boiling components has been deleted.

Hydroxycitronellal ($C_{10}H_{20}O_2$) is one of the widely used perfumery chemicals, having sweet and intense odour, characteristic of linden blossom. It is also known as 7-hydroxy-3, 7-dimethyl-octan-1-al. It has not so far been reported as occurring in nature. According to the International Fragrance and Flavours Association (IFRA), its use in fragrances is restricted due to its possible skin irritation. It is represented by the following structural formula:



Hydroxycitronellal
(Molecular weight 172.27)

Hydroxycitronellal, a saturated hydroxyaldehyde, is suitably stabilized to guard against any appreciable drop in its aldehyde content during storage. The Committee responsible for preparation of this standard observed that 98 percent purity of hydroxycitronellal could be achieved by using better purification techniques hence the purity limit has been upgraded.

For the purpose of deciding whether a particular requirement of this standard is complied with, the final value observed or calculated expressing the result of a test or analysis, shall be rounded off in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*)'. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

Indian Standard

HYDROXYCITRONELLAL — SPECIFICATION

(Third Revision)

1 SCOPE

This standard prescribes the requirements and the methods of sampling and test for hydroxycitronellal.

2 REFERENCES

The following standards contain provisions, which, through reference in this text, constitute provisions of the standard. At the time of publication, the editions indicated were valid. All standards are subject to revision and parties to agreements based on this standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below:

| IS No. | Title |
|------------------|---|
| 326 | Methods of sampling and test for natural and synthetic perfumery materials: |
| (Part 1) : 1984 | Sampling (<i>second revision</i>) |
| (Part 2) : 1980 | Preliminary examination of perfumery materials and samples (<i>second revision</i>) |
| (Part 3) : 1980 | Relative density (<i>second revision</i>) |
| (Part 5) : 1986 | Determination of refractive index (<i>second revision</i>) |
| (Part 6) : 1986 | Determination of solubility in ethanol (<i>second revision</i>) |
| (Part 7) : 1980 | Determination of acid value (<i>second revision</i>) |
| (Part 11) : 1986 | Determination of carbonyl value and |

content of carbonyl compounds (*second revision*)

(Part 19) : 1998 Gas chromatographic analysis of perfumery materials

1070 : 1992 Reagent grade water — Specification (*third revision*)

2284 : 1988 Method for olfactory assessment of natural and synthetic perfumery materials (*first revision*)

6597 : 2001 Glossary of terms relating to fragrance and flavour industry (*second revision*)

3 TERMINOLOGY

For the purpose of this standard, definitions given in IS 6597 shall apply.

4 REQUIREMENTS

4.1 Description

The material shall be a synthetic product, clear and colourless liquid, free from sediment, suspended matter and adulterants, when examined as prescribed in IS 326 (Part 2).

4.2 Solubility

The material shall be soluble in 8 volumes of ethanol (50 percent by volume) when tested as prescribed in IS 326 (Part 6).

4.3 The material shall also comply with the requirements given in Table 1.

Table 1 Requirements for Hydroxycitronellal

| Sl No. | Characteristic | Requirement | Methods of Test, Ref to |
|--------|---|---|---|
| (1) | (2) | (3) | (4) |
| i) | Odour | Persistent sweet, reminiscent of lily of valley | IS 2284 |
| ii) | Relative density ¹⁾ at 20/20°C | 0.915 0-0.924 0 | IS 326 (Part 3) |
| iii) | Refractive index ²⁾ at 20°C | 1.446 0-1.450 0 | IS 326 (Part 5) |
| iv) | Acid value (using 10 g), <i>Max</i> | 4.0 | IS 326 (Part 7) |
| v) | GC Analysis, percent by mass, <i>Min</i> | 98 | Annex A of this standard and IS 326 (Part 11) |
| vi) | Citronellal, percent by GC, <i>Max</i> | 0.5 | do |

¹⁾ The correction factor for relative density for each degree celsius change in temperature is 0.000 50.

²⁾ The correction factor for refractive index for each degree celsius change in temperature is 0.000 38.

5 PACKING AND MARKING

5.1 Packing

The material shall be supplied in airtight and preferably amber-coloured glass, aluminum or any other suitable containers, permitting a minimum of air space, as agreed to between the purchaser and the supplier.

5.1.1 The material shall be protected from light and stored in a cool and dry place.

5.2 Marking

Each container so packed shall bear legibly the following information:

- Name of the material;
- Name of the manufacturer;
- Batch number and date of manufacture;
- Net and gross weight; and
- Flash point.

5.2.1 BIS Certification Marking

The containers may also be marked with the Standard Mark.

5.2.1.1 The use of the Standard Mark is governed by the provisions of the *Bureau of Indian Standards Act*,

1986 and the Rules and Regulations made thereunder. The details of conditions under which the licence for the use of the Standard Mark may be granted to manufacturers or producers may be obtained from the Bureau of Indian Standards.

6 SAMPLING

6.1 Representative samples of the material shall be drawn as prescribed in IS 326 (Part 1).

6.2 Number of Tests

All the characteristics given under col 2 of Table 1 shall be tested on the composite sample.

7 TEST METHODS

7.1 Tests shall be conducted as prescribed in col 2 of Table 1. Reference to relevant standards is given in col 4 of Table 1.

7.2 Quality and Reagents

Unless specified otherwise, pure chemicals and distilled water (*see* IS 1070) shall be employed in tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities, which affect the results of analysis.

ANNEX A

[Table 1, Sl No. (v)]

GAS CHROMATOGRAPHIC ANALYSIS FOR HYDROXYCITRONELLAL

A-1 GENERAL

The chromatographic analysis is given on capillary column. The chromatographic conditions given here are for information and guidance.

A-2 PROCEDURE

A-2.1 The analysis shall be done as per IS 326 (Part 19). The typical chromatograms for hydroxy-citronellal in capillary column with the following chromatographic conditions are shown in Fig. 1 and Fig. 2.

A-2.1.1 Capillary Column — Non-polar

Column : PE-5 (50 m × 0.32 mm × 0.25 µm)
Temperature programme: 100°C to 280°C @ 3°C/min

Initial hold : 2 min
Final hold : Nil
Carrier gas : N₂ — Nitrogen (10 psi)
Injector temperature : 220°C
Detector temperature : 290°C

A-2.1.2 Capillary Column — Polar

Column : BP-21 (30 m × 0.32 mm × 0.25 µm)
Temperature programme: 50°C to 230°C @ 5°C/min
Initial hold : 2 min
Final hold : 2 min
Carrier gas : N₂ — Nitrogen (7 psi)
Injector temperature : 220°C
Detector temperature : 240°C

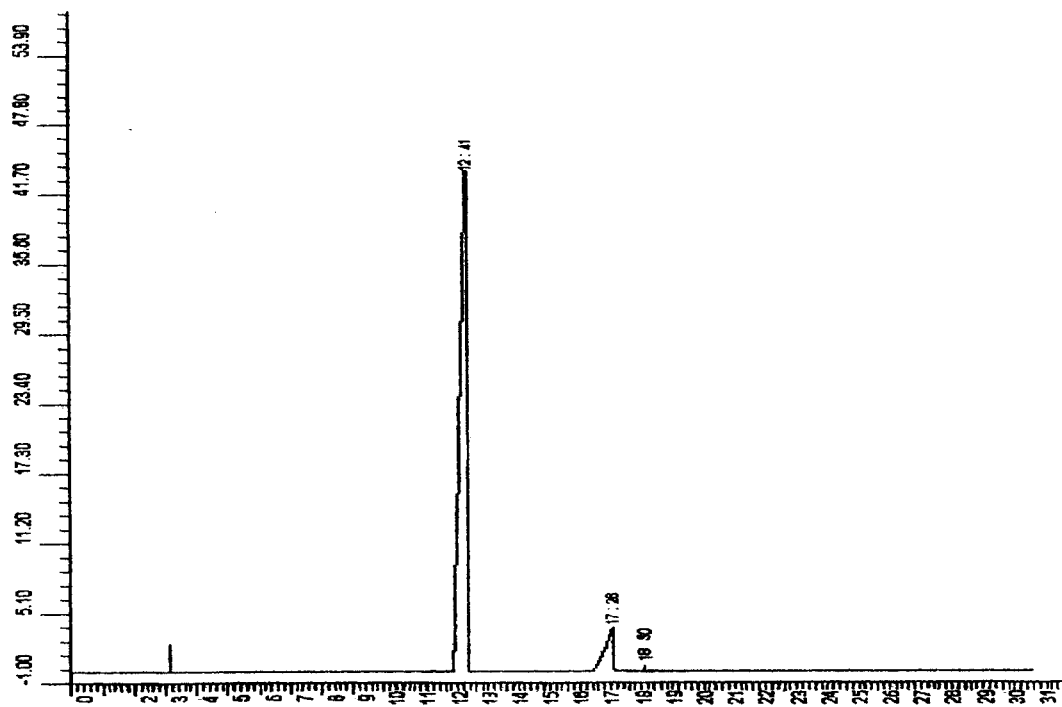


FIG. 1 HYDROXYCITRONELLAL — NON-POLAR COLUMN

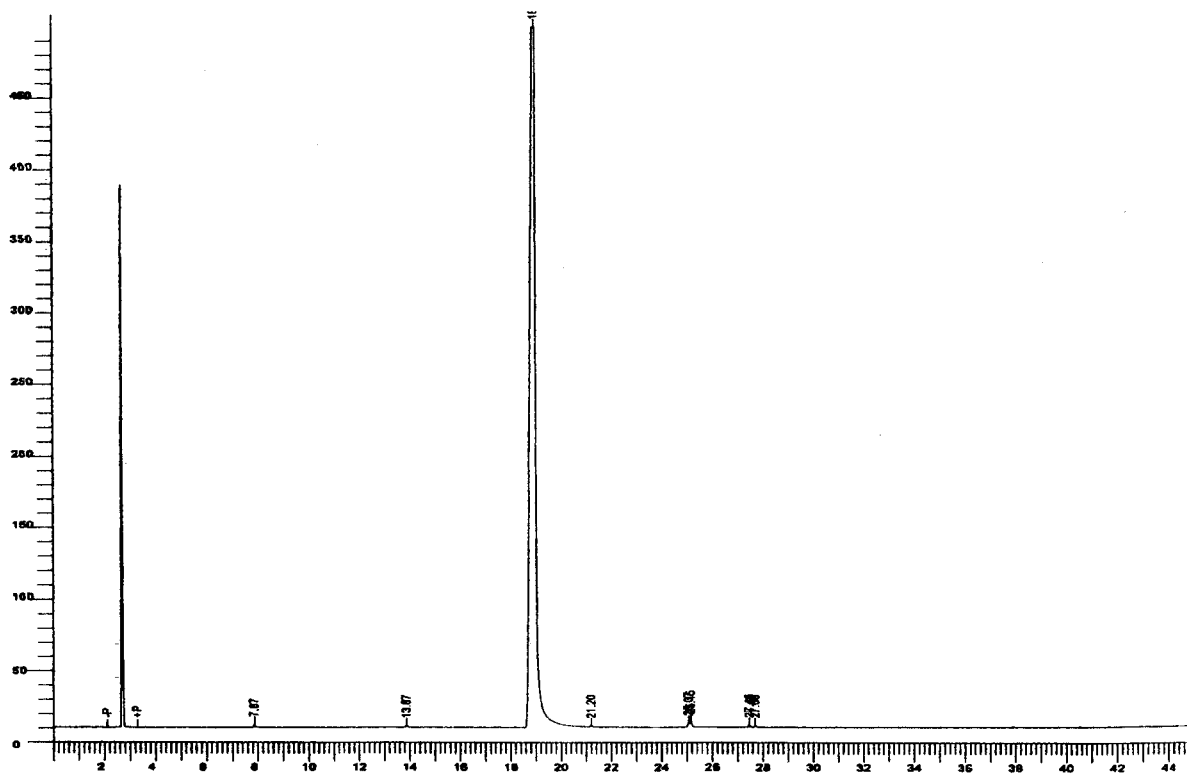


FIG. 2 HYDROXYCITRONELLAL — POLAR COLUMN

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This Indian Standard has been developed from Doc : No. PCD 18 (2251).

Amendments Issued Since Publication

| Amend No. | Date of Issue | Text Affected |
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